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Strength and residual stress of Mg-PSZ after grinding

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Abstract

The influence of grinding with two grinding wheels, differing mainly in diamond-grain size, on the properties of Mg-PSZ was examined. The residual stress, the amount of monoclinic zirconia and the strength of the material were determined. From these measurements depth profiles were obtained for the phase content and the residual stress. The fracture surfaces were examined optically to estimate the critical flaw size. Material ground with “coarse” diamond grains was significantly weaker than that ground with “fine” diamond grains. “Coarse” diamond grains in the grinding wheel resulted in more residual stress and a thicker layer of transformed zirconia than when “fine” grains were in the wheel. This apparent discrepancy is explained with a model based on the occurrence of localized spots of tensile stress beneath the residual stress layer near grain boundaries. These spots are assumed to be larger in number and to contain higher tensile stresses after grinding with “coarse” grains.

1. Introduction

One of the ceramics with good mechanical properties is magnesium-partially-stabilized-zirconia, Mg-PSZ. This is ascribed to the transformation of tetragonal zirconia to monoclinic zirconia [1–6]. One of the main consequences of this toughening mechanism is a fracture toughness, K_{Ic} , of 10–12 MPa m^{1/2} for commercially available Mg-PSZ.

Other consequences of this toughening mechanism are the development of residual stresses and the occurrence of microfracture associated with the transformed particles. The transformation can be caused by surface treatments such as polishing, sawing, grinding or wear. The influence of this surface treatment on the properties of a sample could, for instance, be described in terms of plastic deformation, fracture, phase content, strength and/or residual stress. This influence is usually restricted to a layer located within tens of micrometres from the surface.

Grinding is a reasonably reproducible surface treatment which is often required and can therefore be chosen as the surface treatment to vary. A polished surface can be used as a reference, *i.e.* polishing can be modelled as the treatment which removes material from the surface without causing transformation or microfracture, although it is known that this is only approximately true.

The influence of microfracture on toughness is described in the literature [7–11]. These relations are, however, based on the toughening due to the nucleation and propagation of microcracks caused by the transformation located near a macrocrack. Microcracks caused by a surface treatment such as grinding are present in the material after grinding which means that their influence on toughness and strength is different from the influence of microcracks caused by the transformation. An experimental problem in this area is the difficulty of characterizing microfracture at the surface and, more important still, beneath the surface. Interesting considerations are presented in ref. 12 of a relation between microfracture and the result of strength measurements on two sets of samples containing an increasing amount of stabilized zirconia for one set and unstabilized zirconia for the other set.

The present study was carried out to investigate the influence of grinding with two diamond wheels, differing mainly in diamond-grain size, on phase content, residual stress, strength and fracture behaviour of the material. In particular, the dependence of strength on surface treatment is interesting for obvious reasons. In the literature [13–16], the relation between residual stress and strength is predicted from a theoretical point of view. The present report aims to give some experimental data on this subject and to present a model explaining

an experimentally obtained inverse relation between residual stress and strength.

2. Experimental details

The material, Mg-PSZ from Nilcra Ceramics, Ltd., Northcote, Victoria, Australia, was delivered in tiles of $100 \times 100 \times 10$ mm³. The samples were sawn roughly to the required size. The main characteristics of the material are given in Table 1.

The characteristics of the chosen grinding methods are given in Table 2. The two methods are further denoted by A and B, A being the method with relatively "fine" grains and B that with relatively "coarse" grains. All other adjustable variables and procedures such as wheel dressing and bronze bonding were equal for A and B. The concentration is different, but this cannot

be avoided. This difference in concentration is assumed to be insignificant. These grinding procedures were continued until at least 200 μ m was removed from the sample. In this way all phenomena caused by sawing were removed. The grinding marks were orientated at a right angle to the length-direction of all samples.

Mechanical polishing was done with diamond grains of 2–4 μ m in the last step. Unless stated otherwise no chemical aid was used.

The quantitative phase analysis was performed with X-ray diffraction using Cu K α . The formula used and the derivation of the profile from the integrated values are described in ref. 17.

The residual stress measurements and the phase analysis were performed on batches of bend-strip samples of about $45 \times 10 \times 0.25$ mm³. One surface of 45×10 mm² of each sample was mechanically polished with a chemical aid to ensure the relaxation of all stresses at this surface, until at least 100 μ m was removed. The other surface of the sample was ground according to the defined procedures. The curvature of the sample was measured optically to determine the residual stress [17–19]. These samples, as well as samples from which the ground surface was polished off in various quantities to obtain depth information, were also analysed by X-ray diffraction.

Other sets of samples, used for the strength measurements, were first sawn to an outer shape of about $15 \times 15 \times 5$ mm³. Then about 15-mm long, 1-mm deep and 0.2-mm wide notches were sawn in the samples as shown in Fig. 1. This resulted in ten three-point-bend samples of $15 \times 3 \times 1$ mm³ in a row still attached to their base. The ten surfaces of 15×1 mm² were the surfaces that were stressed during a three-point-bend test. This "cam" shape was chosen to give the surfaces of the ten three-point-bend samples the same treatment without having to face any problems with glue or movement of the samples. The surfaces of 15×1 mm² were ground in accordance with the procedures described before. The ground surfaces of some of these

TABLE 1. Characteristics of the Mg-PSZ. These properties were determined with standard methods

Material	Mg-PSZ ^a
Density (g cm ⁻³)	$5.73 \pm 0.01(7)$
Young's modulus (GPa)	$195.0 \pm 3.0(7)$
Poisson's ratio	$0.324 \pm 0.005(7)$
Vickers hardness (GPa), $P=2N$	12
Mean maximum grain size (μ m)	61
Strength $3pb$ (MPa)	
Polished	$761 \pm 33(10)$
Fracture toughness (MPa m ^{1/2})	
DCB	$10.3 \pm 0.2(3)$
SENB ($3pb$)	$11.5 \pm 1.1(20)$
Roughness, R_a (μ m)	
Polished	0.025
Ground, A	$0.51 \pm 0.08(4)$
Ground, B	$0.69 \pm 0.28(5)$

The experimental methods used are described in ref. 17. The abbreviations SENB and DCB denote single-edge-notched beam and double-cantilever beam respectively.

^aThe numbers given as $x \pm y(n)$ stand for the average x and the sample standard deviation y for n measurements.

TABLE 2. Characteristics of the grinding methods A and B. The main difference between the methods is in diamond-grain size

Grinding methods	A	B
Machine type	Jung	Jung
Grinding type	Circumference	Circumference
Feed in x -direction (mm s ⁻¹)	80	80
Feed in y -direction (mm track ⁻¹)	0.3	0.3
Feed in z -direction (mm)	0.01	0.01
Grinding volume (mm ³)	0.12	0.12
Rotational speed (rotations min ⁻¹)	2800	2800
Cooling liquid	Based on water	Based on water
Diamond-grain size	53–63 μ m	90–106 μ m
Concentration (karate cm ⁻³)	1.32	1.76
Diameter of the grinding wheel	200 mm	200 mm

Fig. 1. Geometry of the samples used. Ten three-point-bend test specimens are still attached to their base. The 15×1 mm² surfaces are after separation of the ten samples stressed in the strength test. The unit of the numbers is mm.

“cams” were removed by polishing until about 1, 2, 5, 10 and 20 μm was polished away in the respective cases. After the required surface treatment the $15 \times 3 \times 1$ mm³ samples were removed from their base by sawing and their strength was measured in a three-point-bend test. With this procedure a strength–depth curve could be obtained with ten measurements for each point on the curve, down to a depth of 20 μm . Additional information from below 20 μm was obtained from single samples from which an amount of material of between 20 and 100 μm was removed. Some of the fracture surfaces were observed by means of SEM to estimate the critical flaw size.

One “cam” was polished until at least 100 μm was removed from the surface to determine the strength of the material assumed to be without any residual stress or microfracture.

Scanning acoustic microscopy was used to examine some of the samples. This was done to detect subsurface phenomena, if possible, without interference from a surface-removing process. The resolution was high enough to visualize grain boundaries.

3. Results

The results from the phase analysis were used to derive the phase content profile. Three X-ray measurements were performed on surfaces which were not polished afterwards. The value for method A is $53.3 \pm 2.2\%$ and for method B $58.5 \pm 1\%$, where $x \pm y$ stands for the average x and the sample standard deviation y for three measurements. These values illustrate the slight but significant difference between the two grinding methods. The measurements performed on samples from which the ground surface was removed through polishing to obtain depth information were single measurements.

The data of the phase analysis are integrated values. These values are used to estimate the monoclinic zirconia depth curve as described in ref. 17. This calculation

results in a shift of the original data as shown in Fig. 2.

The calculated points between 0 and 4 μm for method B were higher than 100%. Since this is physically impossible, there must have been some experimental error in one of the measurements. The interdependence of the calculated values is a consequence of the method used and cannot be excluded. The amount of monoclinic zirconia in the bulk material is initially estimated as 0–15%. This is illustrated in Fig. 2 by the shaded area. A more precise estimate based on some of the experimental results will be given later. The results in Fig. 2 thus show a minimum depth of 20 μm of the transformation zone for method A and 25 μm for method B. These are minimum depths because the precise amount of transformation caused by polishing is not known. All points, except the measurements at 4 μm , indicate more transformation at a certain depth for method B.

The residual stress measurements resulted in a compressive surface stress of 1.02 ± 0.09 GPa for method A and of 1.22 ± 0.10 GPa for method B as determined from three measurements. The average residual stress introduced by method B is thus higher than the stress introduced by method A.

The results of the strength measurements for the first 20 μm are shown in Fig. 3. This figure illustrates the slight but consistent difference in strength after grinding by method A compared with B. Each point on the curve is the average of ten measurements. The strength of samples ground by method A is higher for every depth than that obtained with method B. The steep decrease at 2–4 μm which is present in the phase-content–depth curve, is absent.

In Figs. 4(a) and 4(b) the results of additional measurements made to extend the strength–depth profile to 100 μm are added to the results of the initial

Fig. 2. The calculated amount of monoclinic zirconia as a function of depth. The amount of monoclinic zirconia is given by V_m in percentage. The shaded area indicates the possible amount of monoclinic zirconia in the bulk. $-\Delta-$, Grinding method A; $--+$, grinding method B.

Fig. 3. The strength-depth curve for method A (—Δ—) and method B (—+—) for the first 20 μm which illustrates the consistent difference in strength between the two methods.

(a)

(b)

Fig. 4. (a) The strength-depth curve for method A extended to about 50 μm . (b) The strength-depth curve for method B extended to about 100 μm . Δ , One marker, ten samples; +, one marker, one sample; ---, reference strength.

measurements. The experimental setup for these additional measurements was such that it was not possible to thoroughly control the amount of material polished off the surface. This explains the difference in density

of points shown in Figs. 4(a) and 4(b). It is difficult to estimate the depth at which the strength has decreased to the reference strength, which is the average strength of ten samples with polished surfaces from which at least 100 μm has been removed. This strength is 761 MPa with a sample standard deviation of 33 MPa. This depth appears to be higher for method B than for method A. The depths for both A and B appear to be higher than the 20 μm for A and 25 μm for B respectively, which were estimated as minimal values from the phase analysis. The lack of accuracy is unfortunately inherent to strength measurements on this material.

Examples of critical flaws are shown in Figs. 5(a) and 5(b). The size of these flaws, a maximum length of about 60 μm , is found to be independent of the surface treatment of the sample. This value is comparable with the maximum grain size in this material.

(a)

(b)

Fig. 5. Examples of two fracture surfaces showing a critical flaw of about 60 μm .

Scanning acoustic microscopy visualized only the grain boundaries and showed no features of microfracture.

4. Discussion

There are two extreme approaches in describing aspects of the influence of grinding on a material. One is restricted to the grinding of relatively ductile materials. The deformation of the material is described mainly in terms of plastic deformation [20–23]. Metals are usually used to illustrate principles of this approach. The other is restricted to the grinding of brittle materials. In this case the deformation of the material is mainly described in terms of brittle fracture, and ceramics or glasses are used to illustrate the characteristics of this approach. The material investigated, Mg-PSZ, is a ceramic with an unusually high toughness compared with most other ceramics. Therefore a relatively large influence of plastic deformation can be expected during the grinding of this material.

There is a difference in diamond grain size and concentration between methods A and B. The influence of the difference in concentration on the investigated properties is assumed to be minor compared with the influence caused by the difference in grain size. This difference in size is modelled by a difference in grain shape. The smaller grain of A is assumed to be sharper than the grain of B. The shape of the abrasive is often used as a variable related to the forces working on a sample. A blunt shape results in plastic deformation of materials at higher normal forces. This principle will be used to explain the measured differences in material properties for the two grinding methods.

An earlier study [17] showed the correspondence between residual stress and phase content. The residual stress profile has the same shape as the corresponding phase profile. The absolute stress profile can be derived from the average stress value and the phase profile. The exact amount of monoclinic zirconia in the bulk material is not known because polishing does cause the transformation of some tetragonal zirconia. The derived residual stress profiles shown in Figs. 6(a) and 6(b) are thus dependent on a correction for the amount of monoclinic zirconia in the bulk. An amount of 5–10% monoclinic zirconia for the bulk can be estimated both from Figs. 6(a) and 6(b) and from the result of the earlier study, which shows a residual stress at the surface of 3.6 GPa [17]. Grinding method B introduces a deeper residual stress profile and at most depths a higher value for the residual stress. The main difference between methods A and B is in diamond-grain size. A larger grain thus causes a deeper and higher transformation zone in the material. This is consistent with the general ideas found in the literature [20–23] of a higher normal force during grinding with a larger grain.

Fig. 6. (a) The derived residual stress profile as a function of various corrections for the amount of monoclinic zirconia in the bulk for method A ($-\Delta-$, 0% correction; $-\bullet-$, 15% correction). (b) The derived residual stress profile as a function of various corrections for the amount of monoclinic zirconia in the bulk for method B ($-\Delta-$, 0% correction; $-\bullet-$, 15% correction).

The possibility of fracture originating from the edges of the samples has to be mentioned. Fracture originating from the edges could be due to flaws introduced by the sawing of the samples. However, observations of critical flaws on fracture surfaces gave no reason for the suspicion of a significant influence of failure at the edges.

The interpretation of the strength data presented in Figs. 3, 4(a) and 4(b) is less clear. The strength after grinding by method B is less than the strength after grinding by method A down to a depth of about 25 μm , despite the higher residual stress and thicker transformation zone for method B. This means that the direct influence of the compressive residual stress is not the only phenomenon influencing strength of this material. The apparent discrepancy can be explained with the model illustrated in Fig. 7. Shear stresses, twinning, preferred orientations and other possible phenomena are considered irrelevant to the qualitative aspects of the principles which are used. In this model, grinding introduces the transformation in a surface

Fig. 7. Schematic model of a transformed surface layer and the tetragonal bulk material. The plastic strain corresponding to the transformation results in elastic compressive stresses in the transformed part of the material. The compensating tensile stresses are spread out over the bulk material and can thus be neglected if the structure is perfect, as in 7(a). The compressive stresses can be relaxed near grain boundaries as shown in 7(b). This will cause relatively high localized tensile stresses in the tetragonal structure which can promote fracture at these locations. Further explanation is given in the text.

layer of the material and thus compressive stresses in this layer. This residual stress is developed because the unit cell in the monoclinic structure is larger than the unit cell in the tetragonal structure as illustrated schematically by the spring-like bindings in Fig. 7. A continuum which is transformed at the surface will contain elastic compressive stresses. The compensating tensile stresses are averaged to the bulk of the material. The main inhomogeneities in Mg-PSZ in this simplified model are pores and grain boundaries. These inhomogeneities are locations where the compressive stresses can be somewhat relaxed by strain. Beneath these areas of relaxation, the tetragonal structure will be stretched to an elastic tensile stress as illustrated in Fig. 7(a) and such tensile stresses will promote fracture of the material at these locations. Grain boundaries especially are thus seen as areas where the likelihood of fracture is increased.

Grinding according to the defined procedures results in more residual stress in samples ground according to method B and higher strength of samples ground according to method A. The lower strength of samples ground by B can be explained on the assumption that greater compressive stresses give rise to more areas of tensile stresses and also higher tensile stresses at these locations. These higher tensile stresses will cause larger microcracks beneath the compressive stress layer. These

ideas are in agreement with the results presented in ref. 24. In this article the influence of Young's modulus of a thin coating on the stress state due to a ball indenter at the interface between the coating and substrate is calculated. It is shown in ref. 24 that a stiffer coating, resulting in less strain under a ball indenter, gives smaller tensile stresses at the interface. A stiffer coating as in ref. 24, causing less strain, relates to less compressive residual stress in the present study. A smaller compressive residual stress results thus in smaller stress relaxations, which are equivalent to smaller strains, smaller tensile stresses beneath the compressive layer and thus a higher strength.

To obtain a better indication about the stress state beneath a compressive stress layer near inhomogeneities, a Finite Element Analysis was performed. The analysis used 300 quadrilateral, 8-node, plane stress elements. The geometry used is illustrated in Fig. 8. The dimensions are such that the compressive layer is far thinner than the total thickness, and that the width of the model is large enough to be without influence. The bottom of the model contains also a layer of compressive stresses to avoid curvature of the whole model, without using an extremely large thickness. Stresses were determined along a vertical line for a situation with and without a notch and they were quantified relative to the maximum compressive stress. The notch represents a possible location for compressive stress relaxation, as discussed before. Such a stress relaxation, through a notch or shear or other phenomena, is quite realistic due to a process like grinding. The differences between shear-strain as proposed in Fig. 7(b) and compressive-strain as in this model are not relevant to the principle. The main idea is that there is a possibility of strain, relaxing the compressive stresses, and that larger compressive stresses result in larger strains. The values for the two principal stresses in situations with and without a notch are presented in Fig. 9. It is shown clearly in Fig. 9 that the situation with a notch results in a maximum tensile stress at the interface between the

Fig. 8. Geometry used for the Finite Element Analysis. All dimensions are indicated by a relative number. The vertical line with the x is the line along which stresses are plotted in Fig. 9. See text for further information.

Fig. 9. The two principal stresses S1 and S2 along x from 75 to 100 both for a situation with a notch and without a notch. See text for further explanation. —, No notch, S1; ---, no notch, S2; - · - · -, notch, S1; · · · · ·, notch, S2.

compressive stress and the bulk of the material, of about one third of the maximum compressive stress. The precise magnitude of this stress is not meant to be representative for the modelled situation. It depends on shape of the notch, ratio of thicknesses, etc. The modelling was done to illustrate that a notch in a residual stress layer gives a concentration of tensile stresses beneath this residual stress layer. The calculation does indicate that during a fracture test, fracture is likely to originate from this tensile stressed area.

Additional arguments for the importance of the stress state beneath the compressive residual stress layer can be given. The residual stress of about 3.6 GPa at the surface is significantly more than the failure stress in a three-point-bend test of less than 1 GPa. The absence of a steep decrease in strength at a depth of 2–4 μm , which is observed in the phase-content-depth curve is also an indication of the importance of the subsurface stress state. It is known from observations that fracture of this material is usually intergranular. These considerations are consistent with the concept of areas near grain boundaries with tensile stresses beneath the residual stress layer as one of the strength-determining factors. The bulk material contains flaws varying in shape and size, but the maximum size is the critical flaw size of about 60–70 μm . This size is comparable with the grain size of the material. A logical conclusion is thus that the strength of the grain boundaries in the material, combined with the dual influence of the residual stress, determines the strength of the Mg-PSZ.

5. Conclusions

(1) Grinding with “coarse” diamond grains results in a deeper residual stress layer and a higher residual stress at a certain depth but a lower strength.

(2) The residual stress is modelled to cause localized tensile stresses beneath the compressive stress layer near inhomogeneities such as grain boundaries and pores.

(3) Higher residual compressive stresses at the surface are assumed to result in higher tensile stresses near grain boundaries beneath this compressive stress layer and this will encourage the development of a crack along grain boundaries.

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